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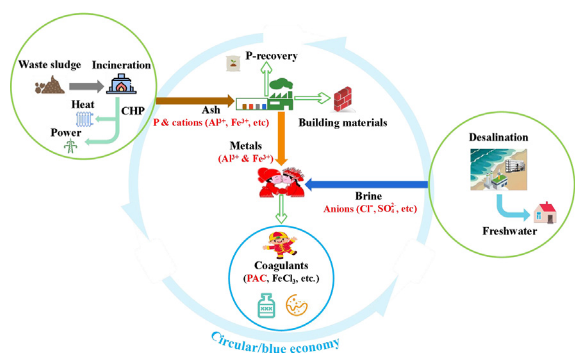
HIGHLIGHTS

- P-recovery from sludge ash is often combined with the removal of metals.
- Desalination produces brine with large amounts of Cl^{-} and SO_4^{2-}.
- Cations and anions from ash and brine can be used to create coagulants.
- An efficient liquid PAC was synthesized from ash and brine by using Al^{3+} removed from ash.
- A circular/blue economy could be established by use of two 'wastes'.

GRAPHICAL ABSTRACT



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ABSTRACT

Sludge incineration and seawater desalination are two approaches that can be used in the disposal of waste activated sludge (WAS) and for obtaining fresh water. As resource recovery from wastewater treatment and water purification is a topic of particular interest in these times, "water mining" has become a focus of research, with phosphate/P-recovery from WAS incineration ash, and extraction of useful elements from the brine of desalination being important steps in the pursuit of a circular/blue economy. However, P-recovery from ash involves removing metals, which need to be disposed of carefully, as does the brine collected. If cations in the ash and anions in the brine could be combined in order to produce coagulants/flocculants, a new circular model would be established. A preliminary experiment for this purpose has demonstrated that a liquid poly-aluminum chloride (PAC) could be synthesized from the aluminum ion/Al^{3+} removed from the ash and the original brine. With this work, we synthesized the liquid PAC by a hydrothermal method, and the results from infrared spectrometer demonstrated that the synthesized PAC was similar to a commercial PAC. Moreover, the synthesized PAC was able to efficiently reduce the effluent turbidity of wastewater treatment plants (WWTPs), especially when compared with the commercial PAC. It is therefore important that research in this area be continued in order to improve the quality of synthesized coagulants and to produce different coagulants based on cations and anions in ash and brine.

1. Introduction

Phosphorus/P is an indispensable nutrient for all creatures on Earth, and plays a key role in material and energy cycles in nature. But it is in limited supply. Urbanization has disrupted the organic cycle of phosphorus (from farmlands to farmlands), largely replacing it with an inorganic cycle of phosphorus, from farmlands to the ocean (Jupp et al., 2021). In order to utilize phosphorus sustainably, P-recovery from wastewater is necessary. This is something that has become a common practice in Europe. Incineration has been identified as an ultimate approach to disposing of waste activated sludge (WAS), and then the incinerated ash is suitable for P-recovery from wastewater-based waste (Hao et al., 2020).

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On the other hand, seawater desalination (SWD) is emerging as a solution for problems faced by regions suffering from water shortage along or near coastal areas. Fresh water produced by desalination has less ecological impact and is an improvement on long distance water diversion from rivers and lakes (Pistocchi et al., 2020). However, brine treatment/disposal from desalination requires careful management, as the brine cannot, generally speaking, be directly discharged into the ocean for ecological reasons. Thus, “brine mining” has also become an important issue in resource recovery.

In practice, P-recovery from ash involves removing heavy metals (copper/Cu, zinc/Zn, lead/Pb, chromium/Cr, cadmium/Cd, mercury/Hg and nickel/Ni) as well as ordinary metals (calcium/Ca, magnesium/Mg, aluminum/Al, iron/Fe, sodium/Na and potassium/K) to obtain relatively pure P-compounds. However, there is also a large number of anions (chloride/Cl^- and sulphate/SO_4^{2-}) present in brine. If cations in the ash and anions in the brine could be suitably combined, coagulants/flocculants might be produced for water and/or wastewater treatment. In this way, two types of “wastes” could effectively be utilized and a new circular/blue economy could be established.

In order to investigate this, an experiment was carried out to produce a coagulant of poly-aluminum chloride/PAC based on both cation (Al^{3+}) from the ash incinerated from WAS and anion (Cl^-) from the brine from SWD. This experiment produced a satisfactory result, in line with expectations of the PAC production.

2. Incineration and ash

Incineration of WAS has been widely practiced around the world for several decades now (Donatello and Cheeseman, 2013). The elemental contents of P, Al and Fe in the ash are sufficiently high, up to 5–10 wt% (11–23 wt% in P_2O_5), 6–18.8 wt% and 2.4–14.5 wt%, respectively (Smol et al., 2020). Wet-chemical process is frequently used for P-recovery from ash, which can produce pure calcium phosphate/Ca-P compounds, struvite and/or phosphoric acid (Luyckx et al., 2020). At the same time, the process can also produce solutions containing high concentrations of Al^{3+}, Fe^{3+} and other metal cations (Smol et al., 2020). Al^{3+} and Fe^{3+} in the ash need to be carefully handled before being disposed of. Thus, utilization of Al^{3+} and Fe^{3+} should be considered first. Among other possible ideas, producing coagulants/flocculants with Al^{3+} and Fe^{3+} in the ash seems to have potential.

The market for both coagulants and flocculants is expected to grow at a compound annual growth rate (CAGR) of 6 % between 2020 and 2025 (Machado et al., 2020). Clearly, the conventional production of coagulants/flocculants will consume a large amount of non-renewable mineral resources. A more environmentally friendly alternative to this would be for Al^{3+} and Fe^{3+} to be extracted from ash and utilized in producing coagulants/flocculants. At present, China is working to expand the market for sludge incineration, and thus supplies of incinerated ash are expected to increase. So it can be expected that coagulants such as PAC and ferric chloride/$FeCl_3$ will be produced with Al^{3+} and Fe^{3+} obtained from ash.

Aluminum phosphate/$AlPO_4$ can be obtained as a product of P-recovery from ash, but it has a very low solubility in water and thus cannot easily be used as a fertilizer. Moreover, Al^{3+} is toxic towards plants. For this reason, Al^{3+} has to be removed from P-compounds by use of techniques such as SEPHOS/SESAL-Phos (Petzet et al., 2011), sulfide precipitation (Franz, 2008), liquid-liquid extraction (PASCH) (Doetsch et al., 2010), ion exchange (Ecophos) (Liang et al., 2019) and complexation with chelating agents (Fang et al., 2018). Among these, SESAL-Phos seems to be particularly efficient. Generally, almost all phosphate radical/PO_4^{3-} and metals in ash can be leached out at pH = 1–2 by use of acidic leaching agents (HCl, H_2SO_4 and HNO_3) (Liang et al., 2019). Increasing pH = 3–4, PO_4^{3-} will result in precipitation in the form of $AlPO_4$ (Petzet et al., 2011). When $AlPO_4$ is dissolved in an alkaline solution (pH = 13), high valent metals are generally insoluble in the alkaline solution (Petzet et al., 2012). Subsequently, dissolved PO_4^{3-} is precipitated with a low-valent metal, resulting in forming a Ca—P compound. As a result, dissolved Al^{3+} remains in the relatively pure alkaline solution, which can then be utilized as a raw material for producing coagulants.

Similarly, Fe^{3+} cannot be dissolved by alkali either, but it can be separated and recovered by organic solvent extraction (Azizitorghabeh et al., 2016). There are many organic extractants, such as tributyl phosphate (TBP), methyl isobutyl ketone (MIBK) (G. Zhang et al., 2015), di-(2-ethylhexyl)phosphoric acid (D2EHPA) and primary amine N1923 etc. (Azizitorghabeh et al., 2016), which can be used for such a purpose.

In short, both Al^{3+} and Fe^{3+} can be easily acquired along with P-recovery from ash, and can be matched with appropriate anions like Cl^- and SO_4^{2-} in order to produce coagulants.

3. Desalination and brine

Seawater represents a potential resource to ensure sustainable availability of water for population and irrigation purposes, especially in some drought-stricken coastal areas of the world (Pistocchi et al., 2020). Reverse osmosis (RO), multi-effect evaporation and multi-stage flash evaporation have become main-stream techniques for SWD, producing large amounts of concentrated brine (Jones et al., 2019). Sustainable management of the brine includes proper disposal and potential resource recovery (brine mining). There are currently 15,906 operational desalination plants, producing around 95 million m^3/d of freshwater and around 142 million m^3/d of brine (Jones et al., 2019). Such a large amount of brine needs to be properly disposed of, or else the marine ecological environment would be affected by brine being directly discharged into the ocean, resulting in increasing temperature, salinity, heavy metals and residual chlorine in local seas (Pistocchi et al., 2020).

In fact, brine is a valuable resource, as it contains originally elements such as K, Na, Mg, Ca, Cl, sulphur/S, bromide/Br as well as trace elements like lithium/Li, uranium/U, rubidium/Rb, iodine/I and even deuterium/D (Lin et al., 2021).

Many approaches to brine mining have been tried, including brine softening, salt production and extracting K, Br, Mg, even Li and U (Lin et al., 2021). Very high concentrations of Cl^- and SO_4^{2-} exist in brine, between 41,829 mg/L and 6050 mg/L, as studies of the Atlantic ocean have shown (Ortiz-Albo et al., 2019). In addition, a large number of studies have been carried out to recover anions from brine. These have included direct nanofiltration (NF) separation of Cl^- and SO_4^{2-} from the brine (Pérez-González et al., 2015), and the separation efficiency of monovalent/multivalent anions from RO brines, which has been investigated by monovalent selective anion membranes used in electrodialysis (ED) (Zhang et al., 2009).

Clearly, Cl^- and SO_4^{2-}-rich brine has considerable potential for producing coagulants with Al^{3+} and Fe^{3+}.

4. Creating coagulants with ash and brine

In principle, Al^{3+} and Fe^{3+} from ash and Cl^- and SO_4^{2-} from brine could be used to produce such coagulants as aluminum chloride/$AlCl_3$, $FeCl_3$, PAC, poly ferric chloride/PFC, poly ferric sulfate/SPFS, poly aluminum ferric chloride/PAFC, polymeric aluminum ferric sulfate/PAFS (Tang et al., 2015), etc. Among them, PAC has an excellent capacity for absorption bridging and net capture for suspended and dissolved substances in water, and is also low on alkalinity consumption and with high stability in a broader range of temperatures and pH levels, due to the presence of different aluminum species (Guo et al., 2022).

There are four processes of PAC production based on raw materials: aluminum-containing mineral, metal aluminum, $AlCl_3$ and aluminum hydroxide/$Al(OH)_3$ methods (Tang et al., 2015). The empirical formula of PAC can be written as $[Al_m(OH)_n(H_2O)_x]\cdot Cl_{3m-n}$ ($0 < n \leq 3$ m) (Guo et al., 2022). As mentioned above, Al^{3+} from the ash and Cl^- from the brine have been used in attempts to produce PAC.

A relatively pure Al^{3+} solution could be obtained from the alkaline solution of the ash (see the above). The brine could be made by

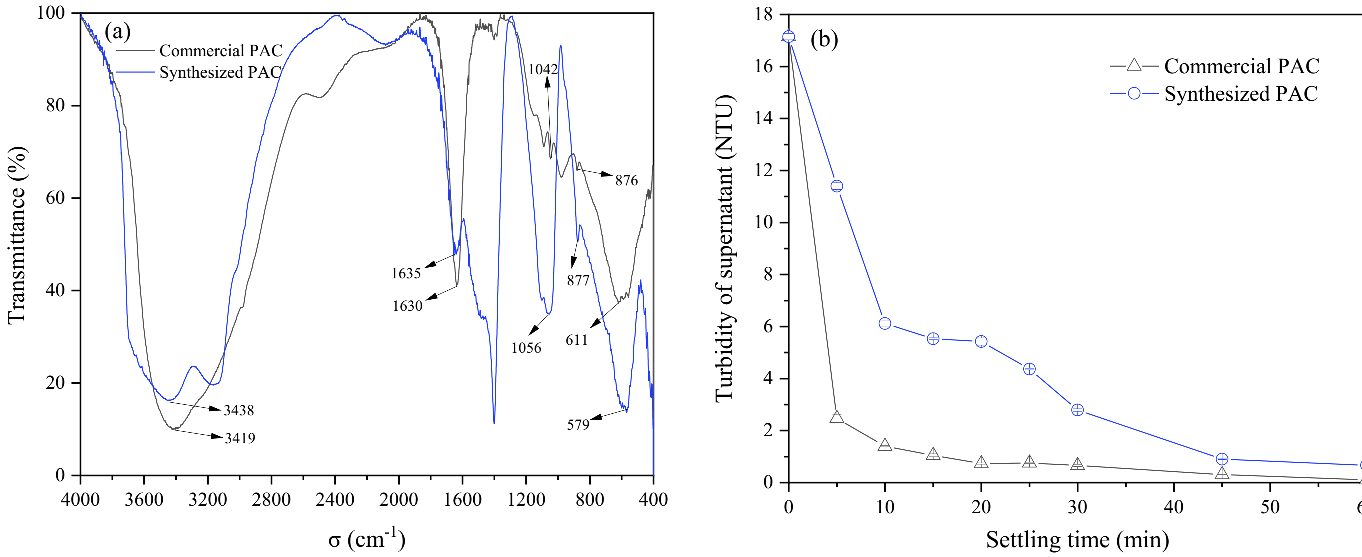

Fig. 1. Comparisons of a commercial PAC with the synthesized product on infrared spectrum (a) and turbidity removal performance (b).

partially evaporating seawater (50 % water content evaporated) and the Al^{3+}-rich solution was directly mixed with the brine, based on a Al/Cl molar ratio of 1:3, and then pH was adjusted to 3.0, followed by heating and polymerizing in a water bath of 70 °C and next by being stirred intensely at 350 rpm for 2 h. Finally, the mixed solution was on standing for maturing at 70 °C in a constant temperature to enhance the polymerization reaction, and then a liquid product of PAC-like was synthesized.

A commercial PAC coagulant was also used as a counterpart so that the performance of the synthesized PAC-like product could be compared, as illustrated in Fig. 1. The liquid PAC-like product was dried and compared with a commercial PAC by the infrared spectrometer analysis (Fig. 1a). The infrared spectroscopy (Fig. 1a) in the range of 400–4000 cm^{-1} reveals that two main absorption peaks were quite similar to each other, with the absorption peaks in the wavenumber of 3600–2800 cm^{-1} being generated by the stretching vibration of -OH linked to aluminum ions in PAC and -OH in the water molecules absorbed by the samples (Tzoupanos and Zouboulis, 2011). In the wavenumber of 1800–1600 cm^{-1}, there were absorption peaks generated by the flexural vibration of -OH and also -OH in the water molecules, indicating that there were hydroxyl groups in the synthesized PAC, which contained both structural water and absorbed water (Guo et al., 2022). The absorption peaks near 1100 cm^{-1} were the stretching vibration peaks of Al-OH-Al, indicating an aggregation state (Y. Zhang et al., 2015). Moreover, the absorption peaks at 876 and 877 cm^{-1} indicated an in-plane flexural vibration of Al-OH-Al, which reflected the bridging and bonding effects of oxygen atoms between aluminum atoms in the synthesized process of PAC, with its intensity reflected the number of Al-OH-Al (Tzoupanos et al., 2009). Al-OH-Al formed multinuclear hydroxyaluminum complexes during the hydrolysis of PAC, which can be used as one of the indicators to evaluate the quality of PAC product. Finally, there were strong absorption peaks at 700–400 cm^{-1}, the overall flexural vibration absorption peak of Al-OH superimposed on the absorption peaks of water molecules, also indicating that the synthesized PAC molecules contained hydroxyl and polymeric aluminum. According to the analysis of the infrared spectrum, therefore, the synthesized PAC compound can indeed be confirmed to be a PAC product.

The two types of PAC were respectively applied for testing the performance in removing effluent turbidity of wastewater treatment plants (WWTPs) (dosage at 50 mg/L), in which synthetic effluent with kaolin suspension (up to 17 NTU) was used. The experiment was conducted at $T = 25$ °C and pH $= 8.0$, which was quickly stirred at 500 rpm for 30 s and then slowly stirred at 100 rpm for 15 min, and the results were shown in Fig. 1b. As shown in Fig. 1b, the result was that the turbidity in the synthetic effluent was reduced from 17 NTU to 1 NTU on a 1-h scale by the two coagulants. Within 10 min of reaction, the commercial PAC demonstrated an excellent capacity for removing turbidity (92 %, down to <2 NTU), compared with the synthesized PAC (62 %, down to around 6 NTU). However, the synthesized PAC can be utilized in treating effluent of WWTPs for the purpose of recycling. According to the standards set for recycled water for non-drinking purposes, a turbidity level of 10 NTU or lower is suitable for use in China. Within 10 min of reaction, therefore, the synthesized PAC demonstrated the same ability to remove turbidity as the commercial PAC.

The synthesized PAC was produced directly with brine in which a high level of salinity existed and such impurities as Na, Ca, Mg and K (Table 1). In the following study, a pretreatment process should be attached to extract pure Cl^- and SO_4^{2-} from the brine and to prevent interference by impurities. In addition, increasing the total Al^{3+} concentration, optimizing pH and the water bath temperature in the polymerization should also be attempted.

5. Conclusions

Based on preliminary experimental analysis, some key conclusions can be drawn:

- Resource recovery can be associated with P-recovery & metals' removal from the ash of incinerated sludge, and from brine from seawater desalination.

Table 1
Chemical composition of the liquid synthesized PAC via ICP-OES.

Al (g/kg)	Na (g/kg)	Ca (g/kg)	Mg (g/kg)	Fe (mg/kg)	K (mg/kg)	Pb (mg/kg)
9.27 ± 0.11	1.61 ± 0.50	0.13 ± 0.02	0.44 ± 0.02	4.13 ± 0.95	15.75 ± 13.64	0.59 ± 0.14
Zn (mg/kg)	Cd (mg/kg)	Cr (mg/kg)	Ni (mg/kg)	Mn (mg/kg)	Cu (mg/kg)	Sn (mg/kg)
0.06 ± 0.02	0.04 ± 0.01	0.05 ± 0.01	6.81 ± 1.19	0.20 ± 0.02	ND[a]	ND[a]

[a] ND: Not detected.

- A liquid PAC can be synthesized with Al^{3+} removed from the ash and the original brine, which demonstrated the ability to remove turbidity in the effluent from WWTPs.
- The synergy of incineration ash and desalination brine could produce coagulants/flocculants which could be substituted for commercial ones in orders to aid in the creation of a circular/blue economy.
- Synthesized PAC could be further purified by extracting Cl^- from brine.

CRediT authorship contribution statement

Xiaodi Hao: Conceptualization, Supervision, Writing - Reviewing and Editing, Project administration. **Xiangyang Wang**: Investigation, Methodology, Formal analysis, Writing - Original draft preparation. **Chen Shi**: Methodology, Resources. **Mark C. M. van Loosdrecht**: Supervision. **Yuanyuan Wu**: Resources.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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